

Masking Water When Using a Laser Particle Counter

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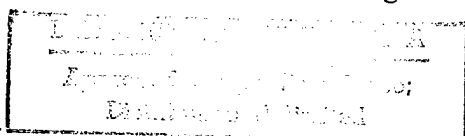
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Abstract: The interaction of a water droplet with the sensor of a laser particle counter causes the water droplet to be measured as a (solid) particle since the droplet obscures at least a portion of the light. Thus when using a laser particle counter both water and solid particles are counted as contaminants. There are advantages for knowing both the concentration of water in an oil sample and the concentration 'solid' particles. Moreover, situations arise when it is advantageous to know the true concentration of solid particles when using a laser particle counter. This paper outlines a method for masking water and obtaining the solid particle content in an oil sample. The paper discusses theory and provides a procedure used for masking water in mineral oils (and also polyalphaolefins, PAO). A 1:1 mixture of mineral oil and mixed solvent (mixed solvent = 3 parts toluene and 1 part isopropanol) effectively eliminates water concentrations up to 10,000 ppm in oil. The mixed solvent essentially dissolves water present while still being soluble in mineral oils. A similar solvent mixture has been found effective in masking water in glycol based oils.

Key Words: Laser particle counter; lubricant contamination; water.

Introduction: The interaction of a water droplet with the laser sensor system in a particle counter causes the water droplet to be measured as a (solid) particle. There are advantages for knowing both the concentration of water in an oil sample and the concentration of 'solids'. However, situations arise when it is advantageous to know the true solid concentration when using a laser particle counter. This paper outlines a method for masking water in the laser particle counter and obtaining the solid content of the oil sample. The procedure should also work with a white light particle counting system. This paper discusses the procedure used for mineral oils (and also polyalphaolefin PAO). A similar procedure has also been found effective in water contaminated polyalkylene glycols (PAGs). It is not known if this procedure is effective with water contaminated polyol esters (POEs), but it will likely be effective.

Water in oil: Water and oil do not mix, but water has a small but finite solubility in mineral oils - we refer to this as dissolved water. At room temperature, the solubility of water is about 20 ppm for a transformer (paraffinic) type oil and 200 ppm for an oil with a large aromatic (naphthenic) content. Dissolved water in mineral oil does not present an interference in a laser particle counter because it is homogeneously dispersed throughout the system. The solubility of



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water in oil approximately doubles for every 25 °C temperature increase. The presence of an additive package will increase the solubility of water in oil as well. Conversely, if the oil cools (after operation) then dissolved water may form free water in the system. The ambient humidity level also affects the amount of water in oil, but this only occurs over long (weeks to months) period of time. Low humidity levels may cause the amount of water to decrease in the system whereas high humidity may increase it.

While dissolved water in oil is relatively benign, free water is almost always deleterious. Free water may be emulsified or non-emulsified. Free water can often be detected by a hazy or cloudy appearance of the fluid. It is free water that obscures light in the laser particle sensor and is therefore detected as a 'particle'. It is also free water that causes numerous problems in oil system machinery. Free water may cause galvanic corrosion between dissimilar metal parts and certain metals (particularly copper and iron), and may promote the corrosion of the system. Copper and iron are particularly good catalysts for oxidizing oils. Furthermore, some metals in the presence of water enhance the oxidation of the base stock of the lubrication oil. Lube oil oxidation products produce precipitates that form varnishes and resins causing poor lubrication and fouling the filtering system. Free water may react (hydrolyze) with antiwear additives such as ZDDP (zinc dialkyldithiophosphates) to form hydrogen sulfide, which ultimately oxidizes to sulfuric acid. Finally, oil and water make a good environment for harboring microbes. Microbes digest and oxidize the oil producing organic acids which cause corrosion.

One simple procedure for determining the presence of water in oil is the crackle test. If the sample crackles when heated above the boiling point of water, it indicates the presence of water. However, this test does not indicate the quantity of water. The industry standard for measuring water in oil is the Karl Fischer reagent method (ASTM D-1744). This method is considered accurate to 10 ppm. One problem with the Karl Fischer reagent is that it reacts with certain additives (for example ZDDP) present in the oil, although procedures exist to circumvent this difficulty. The laser particle counter user may wish to correlate the results using the procedure outlined in this paper with laboratory Karl Fischer water determination. That is determine the amount of water present in the oil sample with Karl Fischer test results, then compare the amount of particles that are eliminated during the masking procedure. The data may be used for screening oil samples and determining which need further laboratory tests, while giving more quantitative results than the crackle test.

Procedure and theory: Since water and oil do not mix, the trick is to find a solvent system that dissolves both water and oil. A solvent like isopropanol (also known as 2-propanol or rubbing alcohol) is miscible (dissolves) in water in all proportions. Unfortunately, isopropanol is not soluble in oil. A solvent like toluene is soluble in oil in all proportion, but does not dissolve very much water. However, toluene is soluble in isopropanol, because both are small organic molecules [1]. Moreover, a mixture of three parts toluene and one part isopropanol is soluble in mineral oils. The toluene is used to dissolve the isopropanol and the mixture becomes soluble in mineral oil, the isopropanol then dissolves any water present in oil. The result is a clear oil solution without the cloudy haziness characteristic of water suspension in oil.

Toluene and isopropanol are cheap solvents available from chemical supply companies. The preferred ratio (3 parts toluene to 1 part isopropanol) is based on laboratory experiments perfecting this technique. A smaller ratio of isopropanol is insufficient for dissolving water, while a larger ratio causes the isopropanol to segregate to the emulsion (that is there is insufficient toluene to dissolve the isopropanol into the oil, so it segregates to the water phase).

A 1:1 mixture of oil and mixed solvent (mixed solvent = 3 parts toluene to 1 part isopropanol) effectively eliminates water concentrations up to 1% (10,000 ppm) in oil. No dilution of the oil sample with kerosene is required. Note that the oil sample containing mixed solvent is considered hazardous waste and must be disposed of properly. A mixture of isopropanol/kerosene or isopropanol/mineral spirits also will dissolve water in oil. However, these two alternate solvent systems are not as effective when the water concentration is large.

The user will have to run a background particle count for the solvent system, and then correct for this background count and the dilution of the hydrocarbon oil when calculating the number of particles in the system.

Apparatus

1. Laser particle counter or white light particle counter.
2. Balance
3. Filtering device with 0.8 micron filter
4. Vacuum chamber for degassing air bubbles (desiccator and vacuum pump is adequate)
5. Beakers and containers

Reagents

1. Toluene
2. 97% pure or better isopropanol (also known as 2-propanol or rubbing alcohol)

Procedure

1. Thoroughly clean and dry the beakers and containers to be used. Remember, containers may be a source of particle contamination.
2. Prepare the mixed solvent. Weigh three parts of toluene (density = 0.867 g/ml) with one part isopropanol (density = 0.786 g/ml). The routine user may wish to prepare a gallon or more of the mixed solvent. Filter the mixed solvent through the 0.8 micron filter.
3. Run the mixed solvent through the particle counter to obtain the diluent background count. If the background count is large clean a new container and filter again.
4. Determine if the oil sample contains free water (frequently a hazy or cloudy oil sample indicates the presence of emulsified or free water). This can be verified with a crackle test. Alternatively, the user can run the oil sample (preferably diluted with kerosene) through the particle counter using standard procedures. If the number of particles at 2 and 5 microns are approximately equal, this may indicate the presence of water.
5. Weigh a 1:1 mixture of oil and mixed solvent. Vigorously shake the oil sample to ensure the suspension of large particles. Degas the oil sample in the vacuum chamber to remove air bubbles. Immediately run the mixed solvent diluted oil sample through the particle counter.

Discussion

Figure 1 is a plot of ISO code versus particle size in an experiment performed using a laser particle counter. The original oil sample (see graph) contained an ISO code cleanliness level of 17 at 2 microns and falls to a code of 10 at 25 microns. The oil sample was spiked with 10,000 ppm water and shows a cleanliness code of 24 at 2 microns and falls to a code of 19 at 100 microns. Note the 'flatness' of the line which indicates the presence of water and saturation of the particle counter. The 10,000 ppm oil sample was treated with a 50:50 mixture of the recommended mixed solvent. The mixed solvent oil sample now shows a cleanliness code very close to the original sample, indicating the effectiveness of this procedure in eliminating water interference in the laser particle counter. We have performed numerous experiments similar to this example that show the usefulness of this technique.

References

- [1] Morrison, R.T., and Boyd R.N., (1973) *Organic Chemistry* Allyn Bacon, Boston, MA

